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Simplified Sampling and Analysis
System for the Determination of Volatile
Organic Compounds in Combustion Effluents

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A SIMPLIFIED SAMPLING AND ANALYSIS SYSTEM FOR THE
DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN COMBUSTION EFFLUENTS

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COMPOUNDS IN COMBUSTION EFFLUENTS

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Abstract

As part of the Resource Conservation and Recovery Act (RCRA), the U.S. Environmental Protection Agency has proposed regulations for owners and operators who treat hazardous waste by incineration. To ensure that these incinerators operate in an environmentally responsible manner, the regulations include a requirement that, for selected principal organic hazardous constituents (POHCs), a destruction and removal efficiency (DRE) of equal to or greater than 99.99% must be achieved (40 C.F.R. Part 264 §264.343). The volatile organic sampling train (VOST) was developed and evaluated for sampling very low levels of volatile organic compounds (VOCs) in incinerator effluents. A simplified system is needed to support engineering projects for waste combustion requiring numerous semiroutine sampling tests. The purpose of this study was to provide a design for a simplified sampling system to address these less stringent requirements. We evaluated sorbents and tandem-bed sorbent sampling tubes to develop a rugged, compact collection system for medium concentration levels of VOCs. We present the development of the system and the laboratory evaluation of the system with selected VOCs.

INTRODUCTION

The Resource Conservation and Recovery Act (RCRA) (1) requires owners/operators of facilities that incinerate hazardous waste to ensure that their incinerators are operated in an environmentally safe manner. The Code of Federal Regulations, Title 40, Part 264, §264.343, requires that a destruction and removal efficiency (DRE) of 99.99% be achieved for each principal organic hazardous constituent (POHC) monitored during a trial burn.

The DRE standard requires sampling and analysis to quantify POHCs in the waste-feed material and stack-gas effluent. The methods manual, "Sampling and Analysis Methods for Hazardous Waste Combustion," provides information on methods that are applicable to process streams of hazardous-waste combustion units (2).

Trial burns at hazardous-waste incinerators suggest that volatile POHCs and volatile products of incomplete combustion (PICs) may be important components in incinerator effluents. The collection and determination of volatile organic compounds (VOCs) boiling below 100 °C have been achieved by using bags, bulbs, and the volatile organic sampling train (VOST) (3). The methods manual provides information on these methods. A protocol for the VOST has been developed (4). The VOST protocol uses multiple sorbent traps with Tenax-GC/charcoal for sampling the stack effluents. Analysis with desorption through a purge-and-trap (PAT) apparatus reduces water input into the chromatographic column. Detection and quantification of the POHCs are performed by mass spectrometry. The VOST thus provides increased sensitivity to low-level concentrations of volatile POHCs because of its ability to concentrate the gaseous effluent. The collection efficiency and analytical sensitivity of the VOST method allow for a DRE of 99.99% when POHCs are present in the waste feed at 100 µg/g or higher.

The purpose of this study was to provide the foundation for a simplified sampling-and-analysis system for medium concentration levels of VOCs. We examined sorbent systems that would reduce the number of sorbent tubes and the complexity of the sampling system. We also examined desorption methods that would eliminate the need for PAT desorption of sorbent tubes. Our objective was to provide sensitivity sufficient to permit calculation of a DRE equal to or greater than 99.99% when POHCs are present in the waste feed at 1000 µg/g or higher.

This report describes the preliminary development of a tandem-bed sorbent system using a single sorbent tube and thermal desorption. PAT desorption is not required in this proposed system. Laboratory evaluations of the sampling-and-analysis system were performed with candidate volatile POHCs.

EXPERIMENTAL METHODS

The primary focus of our experimental work was to evaluate five sorbents and two types of sampling tubes. GC/FID was used to determine the VOCs that were desorbed from the sampling tubes.

Selection of Solid Sorbents

After a review of the literature, we selected five sorbents that have been used successfully in sampling and were prime candidates for use in semiroutine sampling to support engineering projects.

We wanted to evaluate sorbents that may be as effective as or more effective than Tenax-GC for collecting VOCs in the VOST. Thus, we screened five sorbents (Tenax-GC, Sphero-carb, Ambersorb XE-340, Ambersorb XE-347, and Carbo-sieve G) using 3.8 cm of the sorbent material in 3- and 8-mm-OD sampling tubes. A description of these sorbents is summarized in Table 1. In the final sorbent-tube design, we used tandem-bed sorbents in the sampling tubes.

Preparation of Sorbent Tubes

We used two types of sorbent tubes for the sorbent screening. Initially, we used a 3-mm-OD x 11-cm glass tube filled with 3.8 cm of the sorbent. A minimum amount of glass wool was used in each end of the glass tube to hold the sorbent in place. Because of a limited capacity, the 3-mm-OD glass tube was later replaced with an 8-mm-OD glass tube.

Construction of Test-Atmosphere Generator

We constructed a vapor-generator system capable of producing atmospheres containing the following VOCs: benzene, carbon tetrachloride, chlorobenzene, tetrahydrofuran, trichloroethylene, and vinyl chloride. The system utilized permeation tubes (Kin-Tek Laboratories, Inc., Texas City, TX) to provide a constant output of each of the compounds listed above. These permeation tubes consisted of short lengths (10 cm) of Teflon tubing with a liquid phase of the compound sealed inside. Vapor from the tube was carried first into a mixing chamber and then to a sampling chamber with charcoal-filtered air. The concentration of the VOCs in the test atmosphere was controlled by adjusting the flow of dilution air that mixes with the vapor stream in the mixing chamber. The system also allowed for the introduction of humidified air through a separate air stream.

TABLE 1. DESCRIPTION OF SORBENT MATERIALS

Sorbent	Mesh	Surface area, m ² /g	Supplier	Description
Tenax-GC	60/80	19	Supelco	Polymer of 2,6-diphenyl- <u>p</u> -phenylene oxide
Sphero carb	60/80	1200	Analabs	Carbon molecular sieve spheres
Ambersorb XE-340	20/40	400	Rohm and Haas	Synthetic carbonaceous spheres
Ambersorb XE-347	20/40	350	Rohm and Haas	Synthetic carbonaceous spheres
Carbosieve G	60/80	-- ^a	Supelco	Carbon molecular sieve

^aNot available

Design of the Sampling-and-Analysis System

As a result of our preliminary evaluations, we proposed the generalized sampling-and-analysis system shown in Figures 1 and 2. The sampling system is made up of a probe, cooler/condenser, sorbent tube, condensate trap impinger, second sorbent tube (optional), rotometer, and pump. Because the system is intended for short sampling periods, the condensate trap impinger may be considered optional. The analytical system consists of a thermal-desorption unit, gas chromatograph, FID or MS detector, and data system.

Evaluation of Solid Sorbents

We screened five sorbent materials by spiking our prepared sorbent tubes with 4 μ L of a working standard solution. This resulted in the following amounts of each compound in the tubes: tetrahydrofuran--355 ng; carbon tetrachloride--639 ng; trichloroethylene--585 ng; benzene--350 ng; toluene--347 ng; and chlorobenzene--442 ng. Charcoal-filtered air was pulled through the tube at a rate of 0.28 L/min for 3-mm tubes and 0.5 L/min for 8-mm tubes to simulate sampling of standard atmospheres. Each tube was thermally desorbed and analyzed by GC/FID to determine the recovery of VOCs from the sorbent when compared with the direct injection of the standard solution.

Prepared sorbent tubes were used to collect VOCs from an audit cylinder provided by EPA. The cylinder contained low concentrations of vinyl chloride, chloroform, carbon tetrachloride, benzene, and perchloroethylene in nitrogen. We sampled 2.5, 5.0, and 10 L from the audit cylinder at a sampling rate of 0.5 L/min. The sorbent tubes were then thermally desorbed and analyzed using the GC/FID and GC/MS. Concentrations were determined by comparing area responses of the audit cylinder samples to the area responses obtained from calibration standards of VOCs. We prepared calibration curves by spiking 2, 4, 6, and 8 μ L of a standard solution (containing 294 μ g/mL chloroform, 317 μ g/mL carbon tetrachloride, 175 μ g/mL benzene, and 325 μ g/mL perchloroethylene in carbon disulfide) in sorbent tubes.

A vapor-generator system was used to prepare test atmospheres for measuring the capacity of the candidate sorbents. Between 5 and 30 L of a benzene/chlorobenzene (16 ppb each) atmosphere was sampled at a sampling rate of 0.5 L/min. Also, the vapor-generator system was used to produce the mixed-vapor concentrations of the six compounds listed in Table 2. We sampled 2.5, 7.5, and 15 L from the generated atmosphere with each type of sorbent tube at a sampling rate of 0.5 L/min.

To study the effect of humidity on the method, we spiked standard solutions containing six of the volatile organics in 8-mm, 1.9-cm-Tenax/1.9-cm-Sphero carb sorbent tubes. Charcoal-filtered air was pulled through the tubes either with or without subsequent humidification to simulate sampling. In further studies, a sodium sulfate pretube was used to remove moisture. In those studies, the standard was spiked onto the glass wool in front of the



Figure 1. Block diagram of simplified volatile organics sampler.

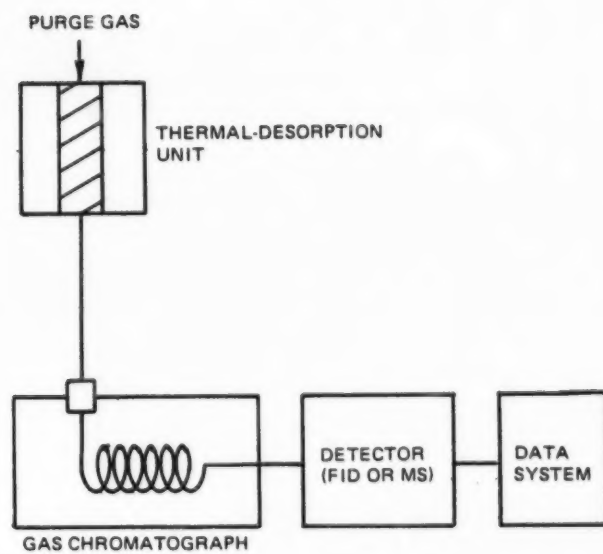


Figure 2. *Block diagram of analytical system.*

TABLE 2. VAPOR CONCENTRATION IN GENERATION SYSTEM

Compound	Vapor concentration	
	ng/L	ppb (v/v)
Vinyl chloride	-- ^a	-- ^a
Tetrahydrofuran	36	12
Carbon tetrachloride	14	2
Trichloroethylene	19	4
Benzene	29	9
Chlorobenzene	30	7

^aCould not determine concentration because there was no available standard for generated concentrations.

sodium sulfate. The filtered and humidified air was pulled through the pretube and the sample tube. Only the sample tube was desorbed for analysis.

Next, we investigated the effect of humidity on the determination of generated-vapor samples. We used the generator to simulate low concentrations of benzene and chlorobenzene atmospheres (16 ppb each) so that we could study the effects of humidity on the sampling method using 1.9-cm-Tenax/1.9-cm-Spherocarb tubes with Na_2SO_4 pretubes. The generator was operated at two levels of relative humidity: 50% and 75%. Samples were collected at a rate of 0.5 L/min, and the sample volumes ranged from ≈ 5 to 30 L.

We also tested the selected sorbent tube for stability. Duplicate tubes were used to sample 7.5 L from the generator at a sampling rate of 0.5 L/min. Then the tubes were analyzed by thermal-desorption GC/FID to establish the vapor concentration in the generator. The experiment was repeated except that the tubes were capped and stored for 3 days at room temperature and then analyzed by GC/FID.

GC/FID Analysis

Initially, we modified the injection port of the HP-5840 GC to allow thermal desorption of 3-mm-OD sorbent tubes. This modification involved diverting the carrier gas through a sorbent tube, which was held in the injection port with an O-ring. Swagelok adapters connect the 3-mm tube to the column inside the GC oven. To analyze standard solutions, a brass tee equipped with a septum was placed in the carrier-gas line. One end of the tee was connected to an empty 3-mm tube leading to the column. Liquid was injected through the septum and swept onto the column by the carrier gas connected to the third end of the tee. Later, we added an external heating block, above the injection port, to allow thermal desorption of 8-mm-OD sorbent tubes. The heating unit consisted of an aluminum cylinder, 5.0 cm long by 4.5 cm in diameter, with a 9.5-mm hole drilled through the center of the face. A Powerstat controlled the desorption temperature. The 8-mm-OD sorbent tube, after it was placed inside the heating unit, was connected on the top to the carrier-gas line and on the bottom to an empty 3-mm tube inside the GC injection port (see Figure 3).

We optimized the GC for the separation of six of the volatile organics: tetrahydrofuran, carbon tetrachloride, trichloroethylene, benzene, toluene, and chlorobenzene. We used a Hewlett-Packard GC/FID. The 1% SP-1000 on Carbowax B (60-80 mesh) 1.8-m x 2-mm glass column was programmed from 60 to 235 °C at 15 °C/min.

Calibration curves were prepared by the direct injection of standard mixtures containing known amounts of the six volatile organic compounds in carbon disulfide. From the calibration data, we calculated the equation for the linear-regression line, slope, and y-intercept.

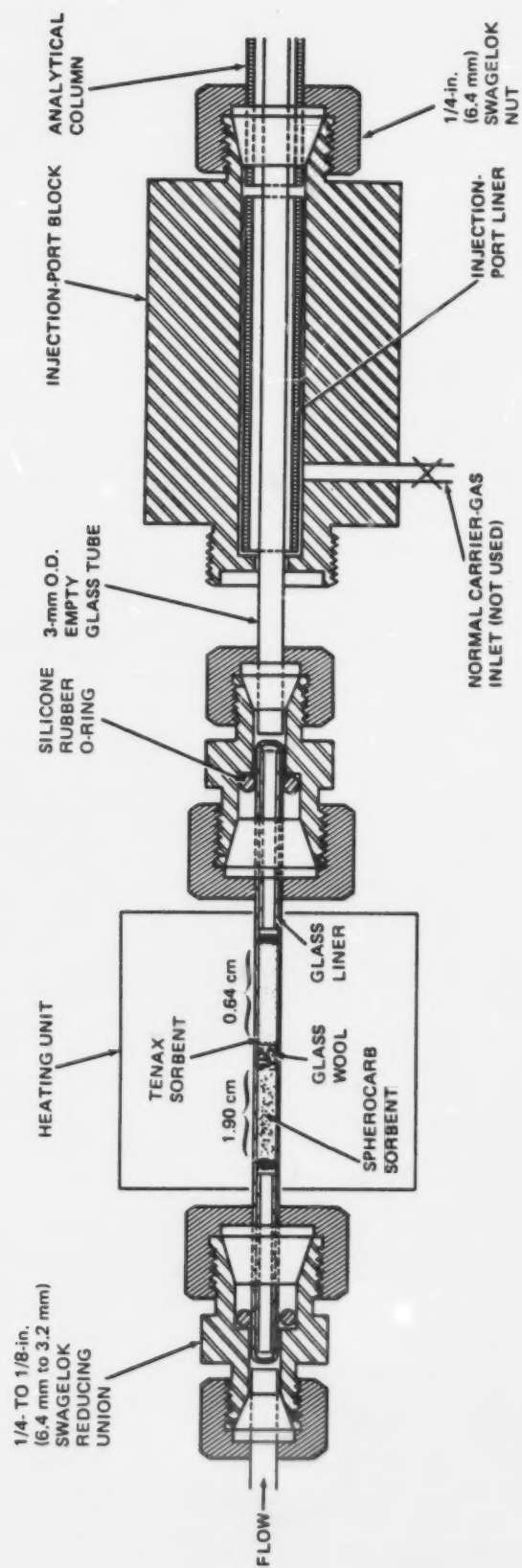


Figure 3. Diagram of thermal-desorption modification for the GC injection port.

RESULTS AND DISCUSSIONS

CALIBRATION

The POHCs chosen for the evaluation of the sampling system were tetrahydrofuran, carbon tetrachloride, trichloroethylene, benzene, toluene, and chlorobenzene. The retention times of selected POHCs were determined by the injection of individual compounds. The GC/FID response to each compound was determined by direct injection of a six-component standard. A representative GC/FID chromatogram of a standard mixture is shown in Figure 4. The linear-regression data for our calibration curves are listed in Table 3. These curves were used to estimate the linear working range for individual compounds to be used in computing recoveries during sorbent screening.

SORBENT SCREENING

Six POHCs were spiked in 3-mm tubes containing 3.8 cm of Tenax-GC. We observed 10-90% breakthrough of the six compounds with 1 min of sampling at 0.28 L/min. After 20 min (5.6 L) only three compounds were recovered: benzene--11%, toluene--35%, and chlorobenzene--69%. Although the 3-mm tube configuration is ideal for direct thermal desorption, it did not have the necessary capacity. Thus we changed to the 8-mm OD tube to allow greater amounts of sorbent to be used in the sample tube.

8-mm Sorbent Tube

The recovery study was repeated with 8-mm-OD sorbent tubes spiked with the same level of VOCs. The recoveries of the compounds from Tenax, Ambersorb XE-340, Ambersorb XE-347, and Spherocarb sorbent tubes are shown in Table 4. Because of the availability of the sorbents and good recovery of standards, we chose Spherocarb and Tenax-GC to prepare tandem-bed sorbent tubes. We prepared 8-mm-OD sorbent tubes packed with 1.9 cm of Tenax (front section) and 1.9 cm of Spherocarb (back section). The recovery data for this mixed sorbent tube are also shown in Table 4. The data indicate good recoveries for all the compounds except carbon tetrachloride, which may be caused by a low and erratic GC/FID response for a low concentration of carbon tetrachloride.

Evaluation of Tenax/Spherocarb Sorbent Tubes

We evaluated three different compositions of tandem-bed 8-mm-OD sorbent tubes containing Tenax and Spherocarb. One sorbent tube contained 1.9 cm of Tenax (80 mg in front section) and 1.9 cm of Spherocarb (250 mg in back section); a second sorbent tube contained 1.27 cm of Tenax and 1.27 cm of Spherocarb; and a third sorbent tube contained 1.9 cm of Tenax and 0.64 cm of Spherocarb.

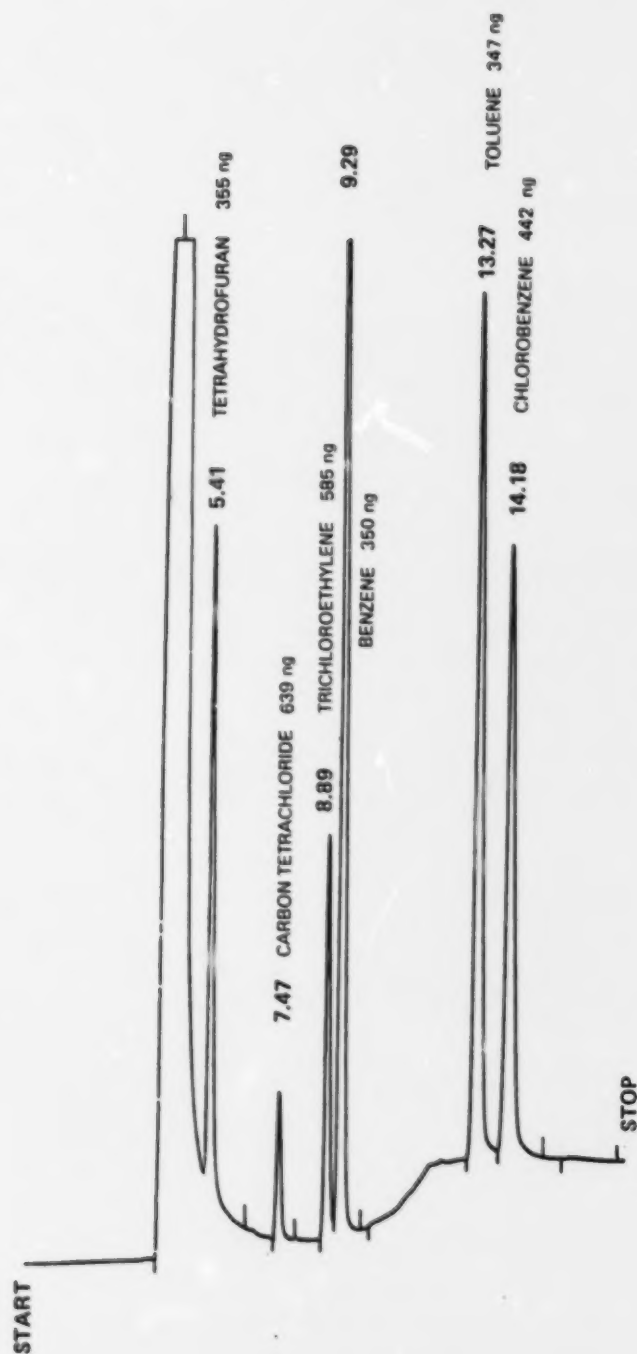


Figure 4. Chromatogram by GC/FID of a standard mixture of volatile organic compounds.

TABLE 3. LINEAR-REGRESSION DATA FOR POHC CALIBRATION CURVES

Compound	Range, ng	Correlation coefficient	Slope, area response per ng	y-Intercept, area response
Tetrahydrofuran	89-532	0.99843	223	5544
Carbon tetrachloride	160-958	0.99995	33.8	196
Trichloroethylene	146-877	0.99998	96.4	634
Benzene	87-524	0.99994	523	2425
Toluene	173-520	0.99960	432	2395
Chlorobenzene	110-664	0.99922	346	5316

TABLE 4. PERCENT RECOVERIES OF VOLATILE ORGANIC COMPOUNDS FROM SOLVENT TUBES

Compound	Volume sampled, L														
	Tenax			Ambersorb XE-340			Ambersorb XE-347			Spherocarb			1.9-cm Tenax GC/ Spherocarb		
	0.8	4.0		1.0	2.3	4.8	9.5	0.9	2.3	4.5	9.0	1.8	2.3	4.5	9.0
Trichloroethylene	103	63		76	70	61	56	98	89	98	83	104	149	108	91
Benzene	103	52		64	24	36	40	105	99	100	104	83	82	79	82
Toluene	85	83		101	100	94	96	113	111	113	114	91	89	87	88
Chlorobenzene	96	93		90	79	61	94	139	118	136	160	101	95	67	85
Tetrahydrofuran	92	13		NA ^a	NA	NA	NA	NA	NA	NA	NA	78	94	78	60
Carbon tetrachloride	109	39		NA	NA	NA	NA	97	109	117	39	85	62	— ^b	91
															81
															— ^b

^a NA = not analyzed.^b "—" = sample interference.

EFFECTS OF HUMIDITY

We spiked a standard solution containing six VOCs in 8-mm OD, 1.9 cm Tenax/1.9 cm Spherocarb sorbent tubes. We pulled clean, dry air through one set of tubes and humidified air through a second set of tubes. A third set of tubes was spiked through a Na_2SO_4 precolumn. Humidified air was then pulled through the tubes, simulating sampling through Na_2SO_4 . All tubes were thermally desorbed, and the recoveries of all analytes were between 88 and 110% when Na_2SO_4 was used as a precolumn. The use of a Na_2SO_4 pretube appears to be one option to eliminate excess water from the sampling-and-analysis system without loss of analyte.

We also sampled from the vapor generator a mixture of benzene and chlorobenzene (26 ppb each) to study the effects of humidity on the sampling method using 1.9 cm Tenax/1.9 cm Spherocarb sorbent tubes with Na_2SO_4 pretubes. The recovery of these two compounds was close to 100%. There was a slight difference in response at the two humidity levels which was probably caused by a difference in the concentrations of the test-atmosphere generator. The humidified air was introduced through a separate valve, and the total dilution air was apparently not duplicated exactly.

STABILITY OF SORBENT TUBE SAMPLES

We tested the stability of six analytes on two Tenax/Spherocarb tube types: a 1.27-cm Tenax/1.27-cm Spherocarb tube and a 1.9-cm Tenax/0.64-cm Spherocarb tube. Duplicate tubes were used to sample 7.5 L from the generator. The tubes were capped and then stored for 3 days at room temperature. Both types of Tenax/Spherocarb tubes gave greater than 85% recoveries for vinyl chloride, tetrahydrofuran, trichloroethylene, benzene, and chlorobenzene. Low carbon tetrachloride recoveries, which were less than 60% on each tube, were probably caused by low response of the FID. The FID response is proportional to the number of oxidizable carbon atoms in the compound. The response diminishes significantly with increasing substitution of halogens in carbon tetrachloride. We chose the 1.9-cm Tenax/0.64-cm Spherocarb tube, shown in Figure 3, as our prototype sampling tube.

ANALYSIS OF A QUALITY ASSURANCE AUDIT CYLINDER

We analyzed the contents of an audit cylinder, Volatile POHC Quality Assurance Cylinder No. 1849 (provided by EPA), using a 1.9-cm Tenax/0.64-cm Spherocarb sorbent tube. The cylinder contained low concentrations of five VOCs in nitrogen: vinyl chloride, chloroform, carbon tetrachloride, benzene, and perchloroethylene. We used the calibration curves obtained from spiked sorbent tubes to calculate the concentrations of chloroform, carbon tetrachloride, benzene, and perchloroethylene in the audit cylinder. We did not analyze vinyl chloride because of difficulties in preparing a suitable low level standard. The concentration values found and the EPA reference values are listed in Table 5. The concentrations found using the tandem-bed sorbent tube agree with the EPA reference concentrations except for carbon tetrachloride. As in earlier experiments this value is significantly lower than the reference values.

TABLE 5. COMPARISON OF AUDIT CYLINDER RESULTS

Compound	Concentration found, ppb	EPA reference concentration, ppb
Chloroform	110	123
Carbon tetrachloride	70	127
Benzene	120	140
Perchloroethylene	125	129

CONCLUSIONS

The volatile organic sampling system developed in this study should be a useful addition to the existing protocols for sampling and analysis of VOCs in incinerator effluents. This sampler is not a replacement for the VOST but is intended to provide a simple method of sampling when extremely low levels of detection for POHCs are not required. It allows the sampling and analysis of compounds boiling below 150 °C with adequate recoveries (45 to 102%) and detection capabilities (LOD 5 to 50 ng). The simplified sampling system consists of a single tandem-bed sorbent tube containing Tenax-GC and Sphero carb sorbents. Direct thermal desorption into a GC/FID or GC/MS provides a rugged and simple sampling and analysis system.

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